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The principle of fibre reinforcement requires load transfer from matrix to fibres. In resin matrix composites it is known, from measurements of mechanical strength, that realisation of load transfer is progressively impaired during water uptake from humid in-service environments. The physical mechanisms responsible for this impairement include the generation of interfacial pressure pockets, the occurrence of which suggests that the optical waveguide behaviour of glass reinforced plastics should be affected

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and might therefore offer a non destructive evaluation technique for monitoring at least one of the causes of mechanical degradation. This possibility has been appraised and experimentally verified.

Two models have been developed and applied to interfacial fracture at pressure pockets attributable to osmosis. Direct photoelastic measurements indicative of load transfer in short fibre composites have been used to test the rates of debonding predicted by the two models.

The net stress across the boundary of the first Wigner cell in a fibre reinforced composite must be zero from which it is inferred that, moving around a fibre, the radial principal stress changes sign several times. The origin of the stress can be differential thermal contraction between fibre and matrix materials during cooling from the resin cure temperature, or inhomogeneous swelling associated with water uptake, or externally applied loads. The number of reversals of sign is determined by the fibre lay-up geometry. In the light of this, interfacial failure is expected to initiate at sites located on the loci of maximum residual radial tension. Also, subsequent propagation of individual pressure pockets is expected to be favoured along these loci rather than around the fibre circumference. The fact that such preferred initiation and propagation are not observed is taken to mean that residual stress is more uniformly distributed than expected. The nature of residual stress generated during resin curing and during water uptake/expulsion has consequently been further investigated.

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# TABLE OF CONTENTS

	Sect ion	Page
	LIST OF FIGURES	5
1.	INTRODUCTION	8
2.	CRP AS OPTICAL MAYEGETDER - NDE OF INTERFACE LIFETIME	10
	2.1 Background	10
	2.2 Specimen preparation	11
	2.3 Experimental results and discussion	11
3.	OSMOSIS IN COMPOSITE MATERIALS	12
	3.1 Hypothesia	17
	3.2 Model 1	13
	3.3 Model 2	15
	3.4 Pheefvations	16
4.	ON THE ASSUMPTION THAT PROXY RESIDE AND TRUE PROMISHIAN STRESS DURING CHRE	17
	4.1 Hypothesis	17
	4.2 Experimental	17
	4.3 Results and Discussion	16
5.	THE INPRVERSIBILITY OF DIMENSIONAL CHARGES IN FROMY RESIDS UNDERGOING IMPARE AND EXPULSION OF MATER	21
	5.1 Previous untk	21
	5.2 Experimental method	21
	5.3 Results	21
6.	REFERENCES	23
7.	PUBLICATIONS, CONFERENCES, SUMMER SCHOOLS	23

#### LIST OF FIGURES

- i. hay diagram for an optical waveguide
- Apporation for mainufacture of (AF wavefuldes 2.
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## 2.2 Specimen preparation

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Polarising microscopy of very low first volume fraction specimens. Figure 4, reveals the occurrence of interfactal pressure packets during the first 200 hours. Suring this time, the resim is also undergoing swelling in order to accommodate the diffused water, figure 5. before saturation, at abound 200 hours, the concentration of diffused water and frace the swelling to thismogeneously distributed and this, in turn, introduces compression against the caternal soffere and tension deeper inside the composite. This offers first as sequelingosed on to that already generated by the differential them. I continue to the differential that already generated that the differential the first differential that are differential that the differential that the materials diffing confirm first season to extremely discontinuous during the initial allows on water replace.

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#### 3.2 Model 1

When formed, each interfacial pocket closely resembles a penny-shaped crack that has grown in a solid tensioned between fixed grips. The overall change in energy is from  $\Gamma_0$ , corresponding to the state of strain energy prior to tracture, to  $\Gamma_0 = \Gamma_0$ , where the Griffith energy  $\Gamma_0$  given by the Zener approximation is

$$L_{i,j} = \frac{1}{2} \left( \frac{1}{a} \frac{2 \cdot 4 \cdot ab^2}{b^2} \right) ab^2 + \frac{3 \cdot ab^2}{b^2} ab^2$$

is the tensile stress applied externally and perpendicular to the crack. It is Young's modules, a and b respectively are the half thickness and ratios of the crack. Seterring to figure 8, it the interfacial cavity could be closed to got attom of traction on its wall, the stress of action of actions the walt, would rise from 1 to 2, and, correspondingly, its valuable would decrease from 1, to 0.

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$$|\psi_{kl}| = \left(\frac{1}{2} + \frac{1}{2} + \frac$$

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In each of the excess of the contestings latter contests with reteriors and attempts. The excess of the latter of the excession of the other contesting pressure, and we fitted all pressure  $\rho = \rho_{\rm p}$  games the same  $V_{\rm p}$  see fixture  $\theta_{\rm p}$ 

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A solute exerts an osmotic pressure equal to the pressure that would be exerted by a gas having the same number of molecules in a volume equal to that occupied by the solution. Consider a change in volume of the interfactal cavity from  $\mathbf{V_0}$  to  $\mathbf{V_1}$  resulting from dissolution of n moles of solute.

$$pV = nRT$$

The strain energy release =  $\int pdV$ 

= 
$$\int_{V}^{nPT} dV$$
  
=  $nRT \log_{e} (\frac{V_{o} + V_{1}}{V_{o}})$   
=  $nRT \log_{e} \frac{\Gamma_{1}}{P_{0}}$ 

 $\rho_{ij}$  and place the values of the osmotic pressure corresponding to volumes  $V_{ij}$  and  $V_{ij}$  respectively.

The overall energy E \* F  $_{\rm gap}$  \* Equation

$$= -\alpha R T \left[ \log_{10} \left( \frac{V_0 + V_1}{V_0} \right) + 3 \frac{F_1 V_1^2}{8\pi ^2 ab^2} \right]$$

This function is elected to Figure 10.

The value of  $V_{\hat{\mu}}$  corresponding to minimum energy release may be found by differentiation

$$\frac{dv_1}{dv_2} = \frac{1}{\sqrt{v_1 + v_1}} + \frac{1}{\sqrt{v_1 + v_2}} + \frac{1}{\sqrt{v_1 + v_2}} + \frac{1}{\sqrt{v_1 + v_2}} = 0$$

$$= 0 \text{ when } v_1^{-2} + v_1^{-2} + 4 + ab^2 \text{ net} = 0$$

We must take the positive root, hence

$$V_1 = -\frac{v_0}{2} + \sqrt{\frac{4ab^2nRT}{3E} \left(1 + \frac{3EV}{16\pi ab^2} \frac{2}{nRT}\right)}$$

$$\sim -\frac{v_0}{2} + \sqrt{\frac{4ab^2nRT}{3E}} \qquad \text{for large values of } b$$

# 3.3 Model 2

Alternatively, interfacial failure due to cracks inflated by osmotic pressure could be modelled as follows. Suppose a constant pressure reservoir supplies the "gas"

Energy, E = 
$$-p_1 V_1 + \frac{1}{2}p_1 V_1 = -\frac{1}{2}p_1 V_1$$
  
=  $-E_{elastic} = -\frac{2\pi}{3} \frac{p_1}{E} \frac{1}{2} ab^2$ 

but  $E_{surface} \sim 2 \gamma \ \pi \ ab$  , where  $\gamma$  is the specific surface energy

so 
$$E_{total}$$
 =  $2\gamma \pi ab - 2\pi \frac{p}{3} \frac{1}{E}^2 ab^2$   

$$\frac{\partial E}{\partial b} total$$
 =  $2\gamma \pi a - \frac{4\pi}{3} \frac{p}{E}^2 ab$   
=  $2\pi a \left(\gamma - \frac{2}{3} \frac{p}{E}^2\right)^2 b$ 

which is negative if

b > 
$$\frac{3E\gamma}{2p_1}$$
2 i.e. if p >  $\sqrt{\frac{3F\gamma}{2b}}$   
 $\frac{\partial E}{\partial a}$ total =  $2\gamma\pi b - \frac{2\pi}{3} \frac{p_1^2}{E} b^2 = 2\pi b(\gamma - \frac{p_1^2}{3E})$ 

and is negative if b >  $\frac{3E\gamma}{p^2}$ 

 $\frac{\partial E}{\partial b}$ total is evidently more negative then  $\frac{\partial E}{\partial a}$ total, so the expression

for  $p_{\mbox{crit}}$  is taken from the former. Taking as trial values E = 3GPa,  $\gamma$  = 1J/m² and b = 10 $\mu m$  ,

$$P_{crit} = \sqrt{\frac{3.3 \times 10^9}{210^{-5}}} = 10 MPa$$

#### 3.4 Observations

It is important to note the distinctive behaviour of pressure filled interfacial cracks under the two extreme types of inflation outlined in models 1 and 2. Model 2 corresponds to the uniform tensile stress type of loading that was treated by Griffith, and gives rise to an unstable critical crack size such that a smaller crack will not grow while a larger one will grow without limit. This critical crack size corresponds to a maximum in the sum of mechanical and surface energies. Model 1 implies loading at the mouth of the interfacial crack (as by the driving in of a wedge) and results in a stable crack size corresponding to a minimum in the sum of mechanical and gas energies.

Thus, model 2 predicts instantaneous interfacial failure if a constant osmotic pressure equal to a critical pressure can be maintained, whereas model 1 predicts interfacial failure at a rate determined by the requirement that each pressure pocket maintains its volume at a minimum energy level.

In Figure 11, the overall rate of interfacial failure is taken as the rate of loss of load transfer index in short fibre composites. The load transfer index is the optical retardation measured through diameters close to the centre of an individual short fibre minus the optical retardation measured through diameters near the ends of the same fibre. It is evident that interfacial failure proceeds at a rapidly decelerating rate such as would be expected if model 2 dominates at the time of initiation but gives way to model 1 during propagation.

According to model 1, interfacial pressure pockets maintain their volumes at values determined by the square root of the number of moles of dissolved solute. It has been observed that solutes can be leached from the fibre in some cases 4, leaving an 'etched' fibre surface, and in such cases the availability of the solute is presumably governed by the rate of diffusion of the solute to the surface of the fibre. The simplest diffusion model predicts dependence on the square root of time, and the combination of these two processes leads to a dependence of the volume of the interfacial pressure-filled cavities on the fourth root of time. The broken line in Figure 11 corresponds to a (time) 1/4 law.

# 4. ON THE ASSUMPTION THAT EPOXY RESINS ARE FREE FROM SHEAP STRESS DURING CURE

#### 4.1 Hypothesis

The accelerated cure reactions that take place during the elevated temperature curing of epoxy resins promote cross-linking and thereby give rise to shrinkage. There are no superimposed dimensional changes attributable to such processes as chain scissioning, as could be the case in polyesters that contain diffused water, or to the release of volatiles, as might be the consequence of condensation reactions in polyamides for example. The shrinkage is assumed to be homogeneous because any tendency to create shear stress is thought to be relieved by viscoelastic flow. This assumption is based on the premise that before it gels, the fluid resin behaves in a Newtonian fashion and is unable to support shear stress.

#### 4.2 Experimental

19mm diameter soda-lime glass cover slips of two different thicknesses were bonded to 1mm thick soda-lime glass microscope slides. The cover slip thicknesses were  $140\mu m$  and  $230\mu m$  respectively and, the epoxy adhesive was Redux 312/5. The cover slips and microscope slides were thoroughly cleaned by ion bombardment before manufacturing the joints. Each specimen was mounted in a specimen chamber so that the free surface of the cover slip was in close proximity to an optical flat. This assembly was then mounted on an optical bench. In order that a uniform temperature distribution was maintained across the specimen, it was mounted on a thick disc of copper. This in turn was held in good thermal contact with the aluminium specimen chamber. Figure 12 is a schematic diagram of the optical components. The space between cover slip and optical flat is made small enough to allow optical interference between light incident upon and reflected from the free surface of the cover slip. Any changes in shape of the interference cavity, due to deformation of the cover slip caused by non-uniform changes in dimensions of the epoxy layer, cause the pattern of interference fringes to change.

Figure 13 shows a sequence of interference photographs recorded during the warm up period of a specimen manufactured with a 140µm thick cover—slip. The time—taken for the specimen to reach the cure temperature—was—30 minutes and a substantial amount of the shape change occurred during this heating-up period. Initially, the cover slip is slightly deformed convex upwards but this soon gives way to a much larger concave deformation, the subsequent development of which is further examined in Figure—14 by creating Moiré patterns between successive photographs of the interference pattern—and the pattern photographed when the specimen had completed—its half hour cure.—The Moiré fringes are the circumferential fringes—each

of which is the locus of points that have suffered identical displacement normal to the joint. The normal displacement of adjacent loci differ by half a wavelength.

#### 4.3 Results and Discussion

Figure 15 shows the normal displacement at different points across a diameter of the same specimen after 5 minutes at the cure temperature, where  $t_0 \approx 30$  minutes is the total time at the cure temperature. The sign of the displacement field was established by applying an identical positive pressure to the cover slip. The curve fits the parabola

$$w = .073 (x - .0095)^2$$

where w is the normal displacement and x is the distance measured from the edge of the specimen

To a first approximation, the deformation of the cover slip can be regarded as identical to that of a circular membrane, rigidly supported at its edge and subjected to a pressure drop (p) across its surfaces. For such a membrane, love has shown that the tourth differential of the normal displacement is a measure of p. The cover slip is thin. If thin enough for Love's analysis to be applied, then

Since the data presented in Figure 15 is described by a parabola, the fourth differential  $\delta^h w/\delta x^h$  and hence the normal stress  $\sigma_{\chi\chi}$  are evidently zero.

Hence it is concluded that the deformation precented in Figure 15 is caused by radial stresses transmitted from the resin to the cover slip and not the stresses created normal to the joint. To check that curing really does cause the parallel sided disc of resin to transform into a concave lens shape, a specimen was manufactured using cover slips for both adherends. As expected, curing caused this sandwich to deform into a double concave lens as might have been produced by the application of edge tractions.

The analogy to the buckling of a thin circular plate simply supported around its edge and subjected to a uniformly distributed edge force applied in the plane of the plate, has been used to estimate the radial stress in the specimen shown in Figure 13.

The relations between the strains, chaptacements and stresses as given by  $Stoker^{(6)}$  are

$$\begin{aligned} & \mathbf{E}_{\mathbf{x}\mathbf{x}} & = & \mathbf{N}_{\mathbf{x}} + & \mathbf{1} & \mathbf{1} & \mathbf{N}_{\mathbf{x}} + & \mathbf{2} & = & \mathbf{0}_{\mathbf{x}} + & \mathbf{n} \mathbf{0}_{\mathbf{x}} \\ & \mathbf{N}_{\mathbf{x}} & = & \mathbf{2} & & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{n} \mathbf{0}_{\mathbf{x}} \\ & \mathbf{E}_{\mathbf{x}\mathbf{y}} & = & \mathbf{1} & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{n} \mathbf{0}_{\mathbf{x}} \\ & \mathbf{2} & \mathbf{1} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} \\ & \mathbf{2} & \mathbf{1} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} \\ & \mathbf{2} & \mathbf{1} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} + & \mathbf{N}_{\mathbf{x}} \end{aligned}$$

where the \$11,0 components of the displacement of a point on the middle surface of the place are demoted by u.v.w respectively.

The middle surface attended timided by the modulus of clasticity is new denoted by  $\sigma_{nn}$ ,  $\sigma_{nj}$ ,  $\sigma_{nj}$  and the middle surface strains by  $\epsilon_{nn}$ ,  $\epsilon_{nj}$ ,  $\epsilon_{nj}$ ,  $\epsilon_{nj}$ ,

Since we assume radial sy: If y, we may just  $c_0 + c_1$  and making the approximation that 2n/2n is sufficiently small relative to  $1/2(2n/2n)^2$  such that it may be neglected gives

$$1/(w^2 + a_{eq} (v - 1))$$
2.747

where v \* Polsson's talio

Figure 16 shows the distribution of stress  $c=\pm c$  obtained using the above equation for the deformation shown in Figure 15.

The above estimate is based on a deformation which is wholly elastic. An alternative estimate, using a simple ejecculartic model, is as follows. The raw adhesive film has a larger thermal expansion coefficient than glass. During heating to the cure temperature, the tesin is free to expand in the thickness direction but radially it has to work against viscosity and its velocity relative to the glass is zero or close to zero at contact, becoming progressively larger at deeper layers. As a consequence, the cover slip is deformed convex upwards. Approaching the cure temperature, the shrinkage associated with the accelerated cure reaction dominates and the cover slip deformation is reversed to the concave shape sketched in Figure 15. An order of magnitude estimate of the implane shrinkage is obtained as follows. The adhesive layer is 150µm thick and 19mm in diameter, so the ratio of unrestricted in-plane shrinkage to thickness shrinkage is

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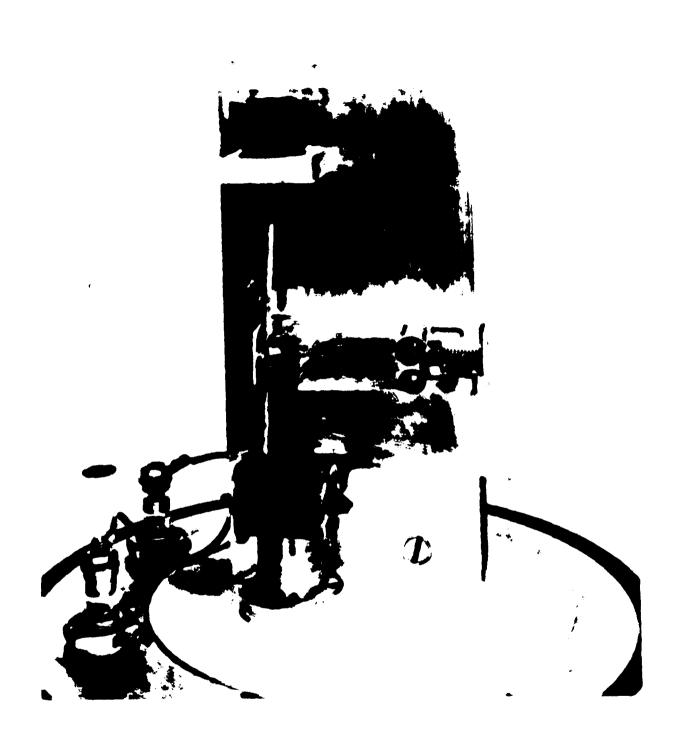
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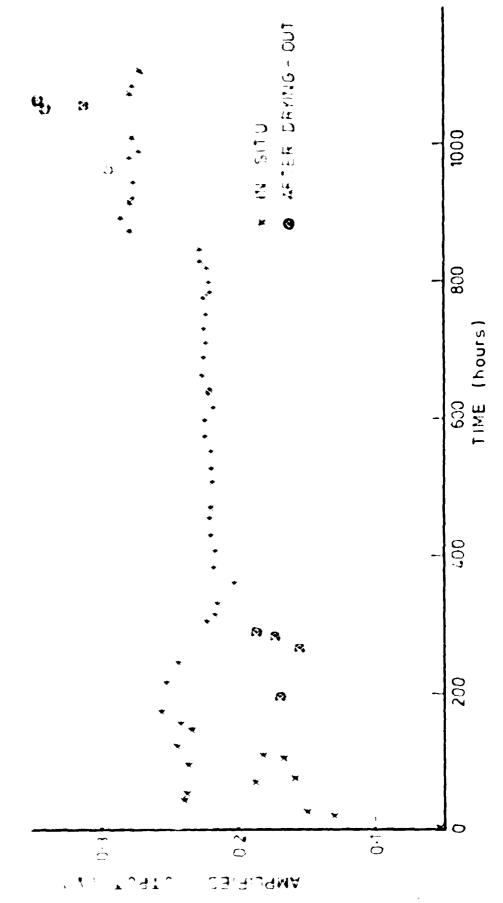
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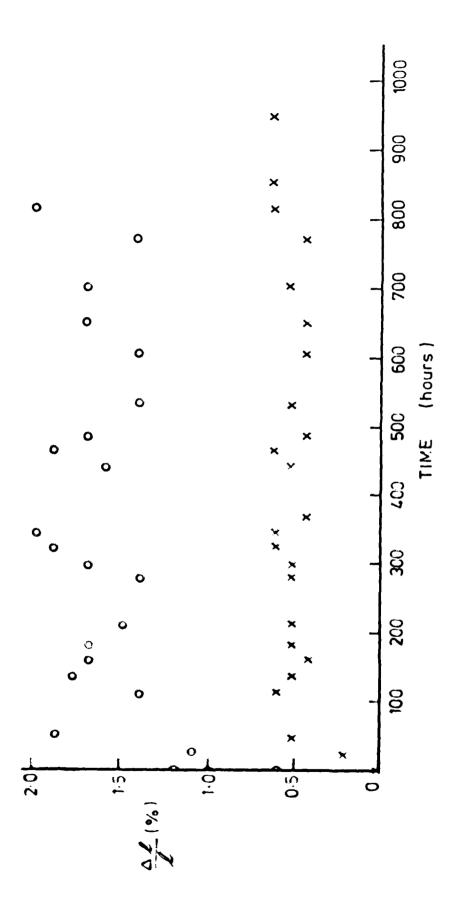
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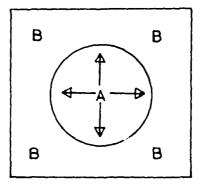
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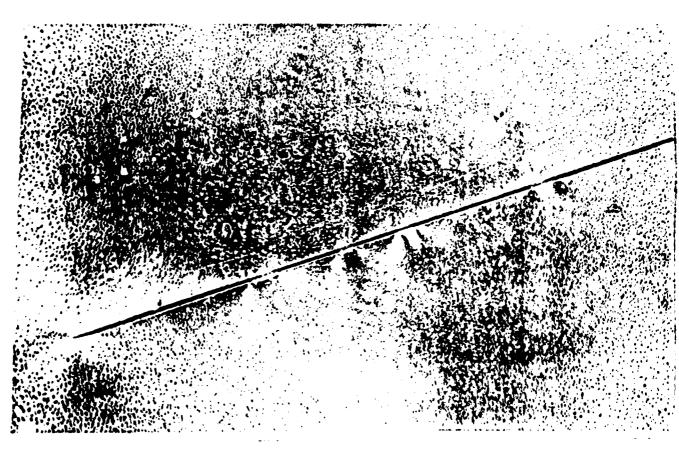
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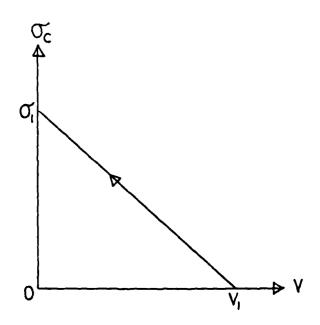
Change in linear dimensions during swelling associated with water uptake at 1600 by two epoxy resin slabs ..



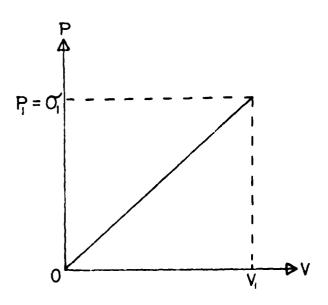
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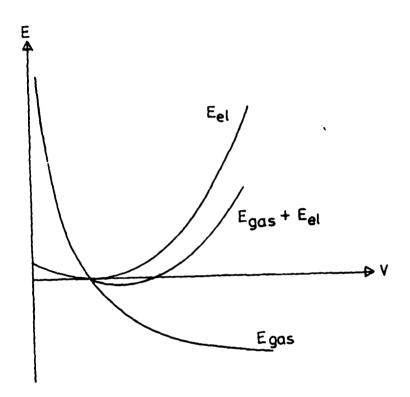
7. Photoclastic contrast caused by pressure pockets on the surface of a graphite fibre in an epoxy matrix composite after 600 hrs. immersion in distilled water at 800.



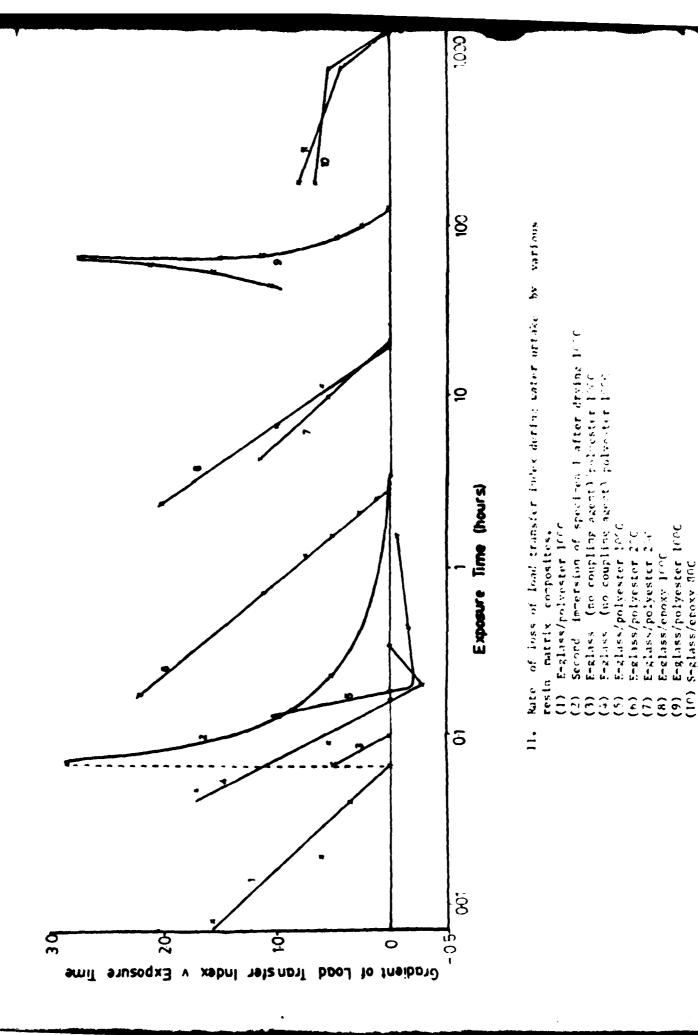
8. Applied stress as a function of volume of a Griffith's crack



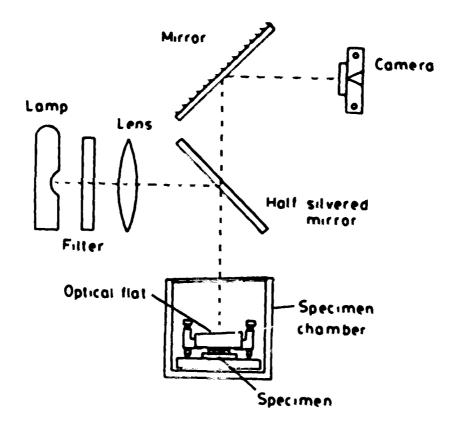
9. Crack volume versus internal pressure for an inflated crack



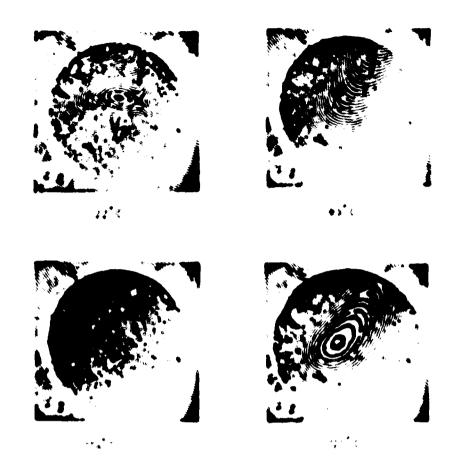
10. Energy versus volume of an inflated crack



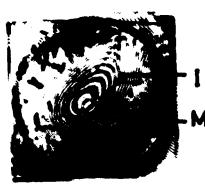
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12. Schematic representation of the optical bench used for optical interference studies of dimensional stability of resins



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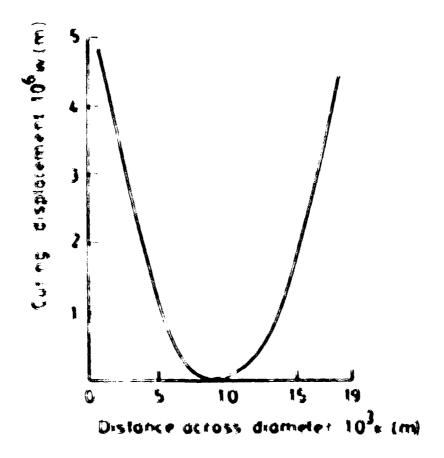


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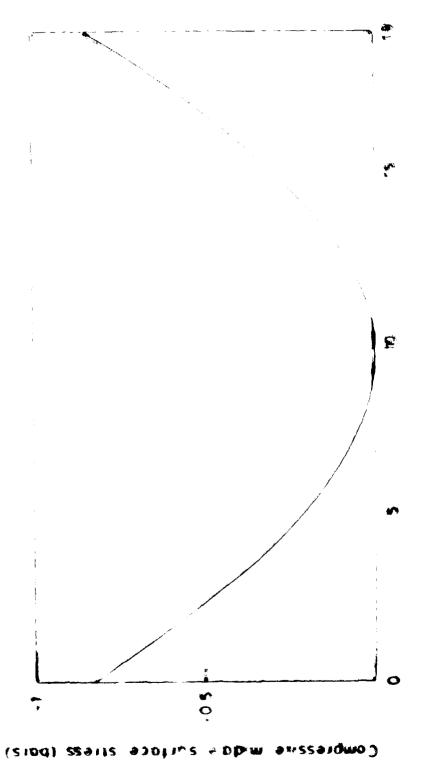




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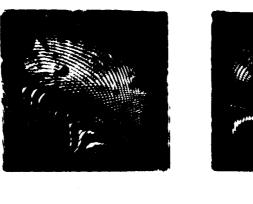


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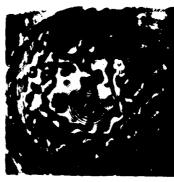


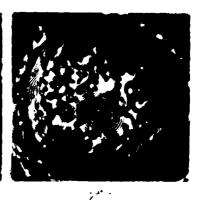




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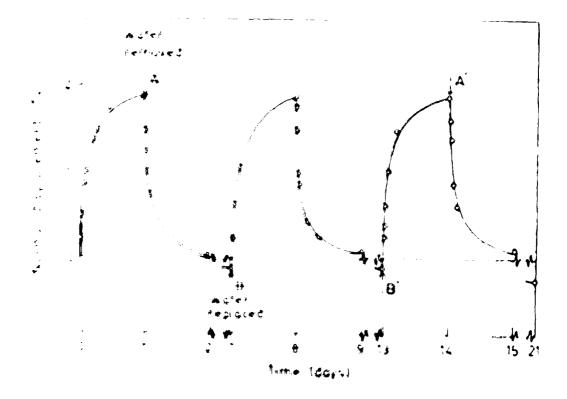




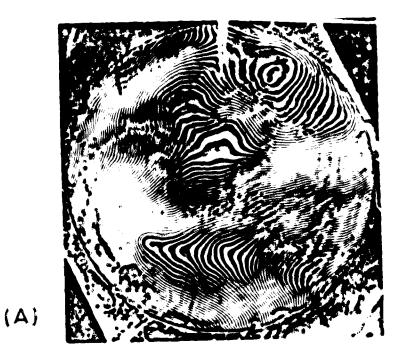


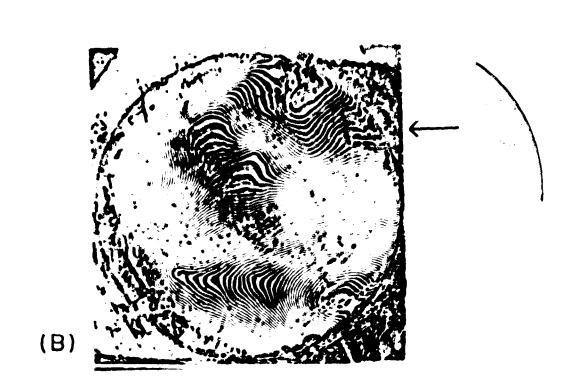


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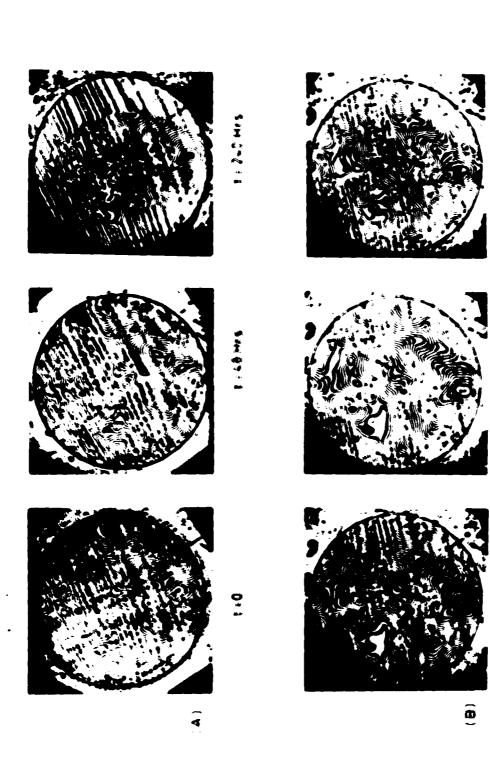


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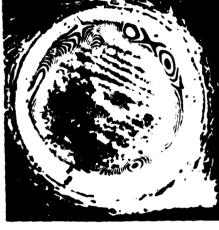






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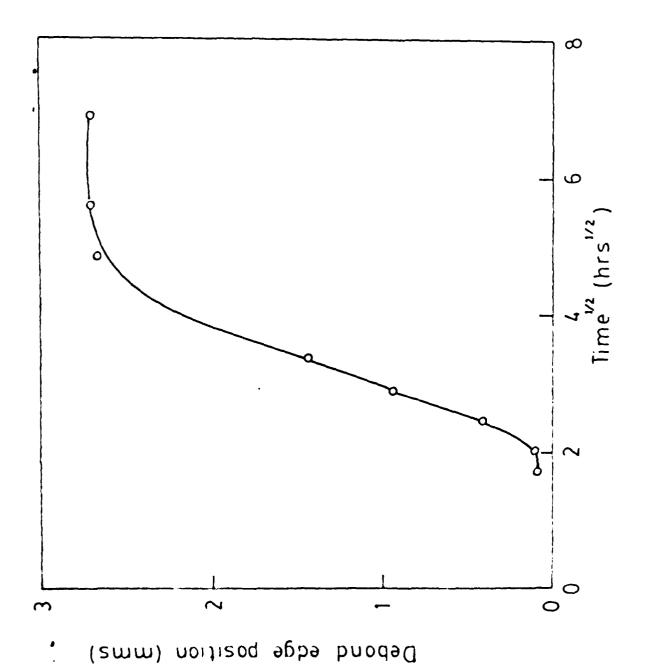


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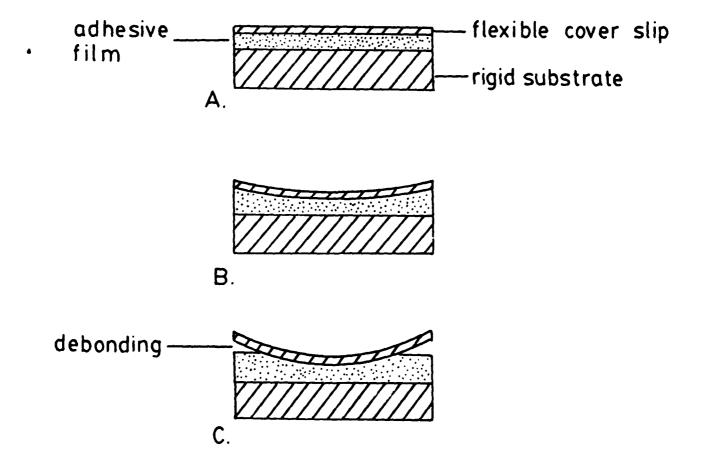
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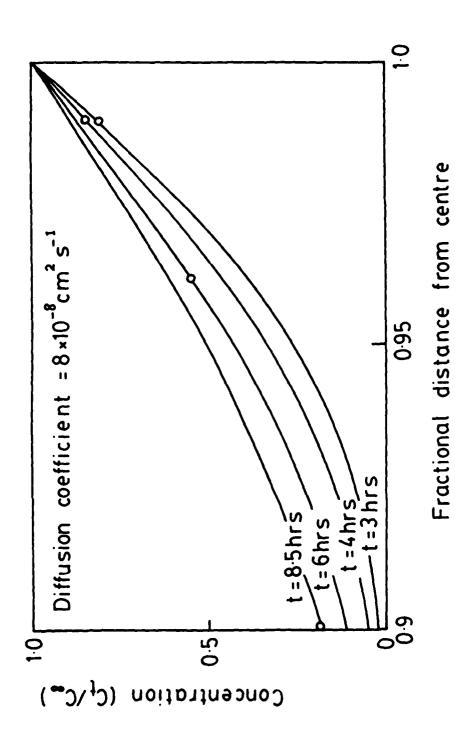
23. Sequence of interference photographs for a FM 1000 specimen incresed



Graph of the position of the debanding edge at 1, the cover slip/adhesive interface plotted as a function of time 2 for the specimen shown in Figure 23 24.



25. Schematic diagram to illustrate the changing geometry of the cover slip during swelling of the adhesive in a joint exposed to an aqueous environment. Debonding follows saturation of swelling at the rim of the joint



Graph of the predicted water concentration  $(C_{\rm L}/C_{\rm w})$  as a function of fractional distance from the centre of the specimen. 0, points representing to critical concentrations for positions ggresponding to the debonding edge shown in Figure 25. After Kinloch  $\{g\}$ , 26.

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